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L27: Entry 2 of 8

File: USPT

Jun 30, 1998

DOCUMENT-IDENTIFIER: US 5773383 A

TITLE: Method of making solid acid catalysts with metal cores

Detailed Description Text (57):

2.3.1. Metallic aluminum reacts at ambient or reflux temperatures, with substances selected from the group of aliphatic or aromatic amines, such as: guanidine, aniline, alkyl-anilines, etc., having at least one unsubstituted H atom at the amine function. Aluminum amides are produced, with hydrogen release. By calcination in air, at 150.degree.-500.degree. C., the layer of amide is transformed into an adherent layer of oxide.

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L27: Entry 3 of 8

File: USPT

Nov 12, 1996

DOCUMENT-IDENTIFIER: US 5573991 A

TITLE: Preparation of metal carbide catalyst supported on carbon

Abstract Text (1):

A process for forming a supported metal carbide catalyst, for example, a Group VIB transition metal carbide, such as tungsten carbide, which process comprises the calcination of a carbon support that has been impregnated with a metal carbide precursor comprising a water soluble salt of: (1) a cation comprising nitrogen-hydrogen bonded moieties, such as a guanidine cation; and (2) an anion, such as a tungstate anion, comprising metal-oxygen bonded moieties, so that upon calcination the product formed is the metal carbide and the by-products comprise ammonia and carbon dioxide.

Brief Summary Text (4):

Pending U.S. Ser. No. 156,670, filed Nov. 23, 1993 teaches that catalytic metal carbide compositions can be formed by the calcination of a guanidine compound, derivative or adduct with a transition metal salt containing the desired metal component of the carbide.

Detailed Description Text (2):

This invention relates to a novel process for forming a supported metal carbide catalyst involving the calcination of a carbon support material which has been impregnated with a water soluble precursor for the metal carbide. The precursor is made by a one-step chemical reaction between a transition metal-containing compound and a carbon containing compound which is low in carbon content as described and claimed in U.S. Pat. No. 5,451,557 which is mentioned hereinbefore. In the broadest embodiment of the invention, the precursor used in the process is a water soluble salt of: (1) a cation comprising nitrogen-hydrogen bonded moieties with a high nitrogen to carbon content; and (2) an anion comprising metal-oxygen bonded moieties, so that upon calcination the product formed is the metal carbide and the by-products comprise ammonia and carbon dioxide. The precursor contains both a metal source, such as the Group VIB transition metal, tungsten, and a carbon source, such as a guanidine compound. Guanidine carbonate, which is of the formula (CN.sub.3 H.sub.5).sub.2 H.sub.2 CO.sub.3, contains only 20% carbon and is an example. The precursor is very soluble in water. Its high solubility allows for the preparation of carbon supported catalysts in accordance with the present invention by impregnation. It allows the use of less volume of a solvent, not exceeding the pore volume of the carbon support that will be impregnated with the solution. This process, which is known as the "incipient wetness method", is preferred for the manufacture of such supported catalysts. The incipient wetness method requires that the volume of the solution be equivalent to the pore volume of the carbon support. Upon calcination of the impregnated carbon support, equal dispersion of the active component on the support will result. High solubility of the precursor will also allow increasing the metal loading on the carbon support at will.

Detailed Description Text (5):

This calcination step does not involve carburization. It is a chemical reduction of the metal ion with the carbon-nitrogen ion of the same compound or from the ammonia released thereafter. It is believed that ammonia will reduce the tungstate ion into a lower oxidation state, which will in situ chemically react with the

carbon in the same molecule forming carbides. The result is a metal carbide containing substantially no excess carbon, which is well dispersed over the carbon support, giving a high surface area catalyst. The ratio of the guanidine to the metal was found to be important for forming a metal carbide suitable for use as a catalyst for reactions such as isomerization of n-heptane. For example, it will be shown later that if the ratio of guanidine to the metal is less than three, other phases such as nitridic or metallic phases will be the main component. These phases will result in the undesirable cracking of heptane to lower hydrocarbons. When the ratio is 3:1, the only phase would be W.sub.2 C. The solution of guanidinium tungstate would also be easily impregnated into the porous support in one step, then calcined at an industrially reasonable temperature not exceeding 800.degree. C.

Detailed Description Text (7):

A composition of matter which is formed by the solid state reaction of ammonium metatungstate and guanidine carbonate at 100.degree.-200.degree. C., was found to be: (1) completely soluble in water; (2) decomposable at 228 .degree. C., which is different from decomposition temperature of the reactants; (3) contains 35-50% tungsten; (4) contains 5-10% carbon; (5) contains 20-30% nitrogen; (6) has a characteristic X-Ray diffraction pattern not found before; (7) forms mainly tungsten carbide, W.sub.2 C, containing substantially no free carbon, when heated at 600.degree.-850.degree. C. under nitrogen, such carbide showing catalytic activity in chemical hydrotreating reactions known to occur with noble metals, such as platinum and palladium; and (8) aqueous solutions of composition are dry impregnable by the incipient wetness method into a carbon solid support, in accordance with the present invention, prior to calcination, making it possible to produce such supported metal carbide catalysts.

CLAIMS:

5. A process for forming a metal carbide catalyst which comprises the calcination of a carbon support that has been impregnated with a metal carbide precursor which comprises a water soluble salt of: (1) a guanidine cation; and (2) an anion comprising the metal and oxygen, so that upon calcination the product formed is the metal carbide and the by products comprise ammonia and carbon dioxide.

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